

Los Alamos National Laboratory
Environmental Restoration Program
Standard Operating Procedure

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Operation of Siemens X-Ray Diffractometer

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OPERATION OF SIEMENS X-RAY DIFFRACTOMETER

1.0 PURPOSE

The purpose of this procedure is to describe the methods, procedures, and documentation used to obtain X-ray powder diffraction data from the Siemens D- 500 X-ray powder diffractometers.

2.0 SCOPE

2.1 Applicability

This procedure applies to all personnel who are conducting ambient and controlled-temperature or atmosphere X-ray diffraction analysis of any rock or mineral for the Environmental Restoration (ER) program using the Siemens D-500 X-ray powder diffractometers.

2.2 Training

Los Alamos National Laboratory policy requires that all persons using this equipment have completed the Laboratory analytical X-ray safety class within the required time period, and be trained by the machine custodian on the proper operation of the equipment, safety features, and emergency procedures. All personnel using this procedure must document that they have read and understood this procedure, and the seven procedures in Section 1.0, General Instructions.

3.0 DEFINITIONS

Machine Custodian: The person who is in charge of an instrument and is responsible for the maintenance and safety of the instrument. This person's name shall be posted on the front of the diffractometers.

4.0 BACKGROUND AND/OR CAUTIONS

4.1 Principle X-ray diffraction is a method by which investigators can identify the minerals present in a rock and obtain quantitative information on their abundances and physical properties.

4.2 Malfunctions of the equipment are readily apparent. For the diffractometers, one either obtains counts or no counts. Failure of the TTK and ITC4 units during controlled temperature runs is readily apparent by monitoring the sample temperature displayed on the ITC4 controller.

4.3 The diffractometers produce ionizing radiation using high voltage sources. However, the diffractometers are interlocked such that if the panels are all in place, risk to the operator is almost non-existent.

4.4 Only two possibilities (rare) for data corruption exist: 1) loss of X-ray flux; 2) occurrence of noise spike. The first problem can be identified by the lack of any diffracted intensity, i.e., counts = 0. Such data should be discarded. The occurrence of the second possibility can be noted by "peaks" only one 2θ step wide. Such data are generally usable and only need to be discarded when a noise spike overlaps on an important peak.

4.5 During a variable-temperature X-ray run, data shall be rejected if, after a run is completed and the TTK attachment is opened, the sample is found to have fallen or blown (due to vacuum) out of the sample holder.

4.6 The dated entry on the Daily Activity Log (see SOP-01.04), for each X-ray run shall constitute evidence that the procedure has been implemented and satisfactorily accomplished for the X-ray run.

5.0 EQUIPMENT

- Siemens D500 X-Ray Powder Diffractometers
- DIFFRAC5000 - Siemens Commercial X-Ray Data Package
- Anton Paar TTK Temperature Attachment. (This attachment is capable of controlling the sample temperature between liquid nitrogen (- 195.8°C) and ~300°C under a vacuum)
- Oxford Instruments ITC4 Temperature Controller
- Roughing Vacuum Pump

6.0 PROCEDURE

The machine custodian is responsible for both alignment and calibration of the diffractometers and the training of any potential users of the diffractometers. The principal investigator (PI) has the responsibility to assure correct implementation of this procedure for ER work. The PI may delegate performance of the procedure to any properly trained and certified individual.

6.1 Calibration

For Alignment and calibration, see Calibration and Alignment of the Siemens Diffractometers, SOP-09.04. Temperature calibration of the TTK attachment shall be performed at several temperatures, preferably at liquid nitrogen temperature and in boiling water, as outlined in reference D. Calibration of the sample temperature may be performed using an independent temperature measuring device such as the thermocouple probe operated in accordance with TWS-EES-DP-105, Thermal Calibration Procedure or using known melting point(s) of a crystalline phase.

6.2 Control of Samples

6.2.1 Samples will be tracked, stored, shipped, and handled in accordance with the procedure for Sample Identification and Control for Mineralogy-Petrology Studies, (see reference H2), SOP-01.03, Handling, Packaging, and Shipping Samples, and SOP-01.04, Sample Control and Field Documentation. Great care shall be exercised to label all X-ray runs with correct sample name, checking sample output against names on the sample bottles.

6.3 Diffractometer Operation

6.3.1 Turn on diffractometer as outlined in the operating instructions (Ref A).

6.3.2 Insert sample and turn on X-rays either by pressing the shutter-open button or by placing shutter in automatic mode.

6.3.3 The instrument is operated and data analyses conducted using the Siemens software package, DIFFRAC5000. Refer to the DIFFRAC5000 users manual (Ref B).

6.3.4 If the samples are to be X-rayed at variable temperatures using the Anton Paar TTK attachment, the TTK attachment is operated and maintained as outlined in the TTK instruction manual (Ref C), and the Oxford ITC4 temperature controller is operated as outlined in the ITC4 Operators Manual (Ref D)

6.4 System Shut-Down

The diffractometer is shut-down in accordance with the instruction manual (Ref A).

6.5 Data Analysis

6.5.1 The data are regressed and displayed using the Siemens DIFFRAC5000 software package.

6.5.2 Crystalline phases are identified by comparing their patterns with patterns of pure standards, patterns from the JCPDS files (Ref E, F, G), or with calculated patterns.

6.5.3 Quantitative X-ray diffraction analysis is conducted in accordance with SOP-09.03 and TWS-EES-DP-116 (Ref H3).

6.6 Procedural Deviations

Deviations from this procedure shall be fully documented in the Daily Activity Log in SOP-01.04, explaining the deviation and the effects it may have on the resulting work.

7.0 REFERENCES

- A. Siemens D500/501 Operating Instructions, C72000-B3463-A42, Siemens Corporation, Cherry Hill, New Jersey.
- B. Siemens DIFFRAC5000 User's Manual, Siemens Corporation, Cherry Hill, New Jersey.
- C. Anton Paar Instruction Manual for TTK-Temperature Attachment for Low and Medium Temperature, Anton Paar KG., Karntnerstrasse 322, A-8054 Graz/Austria.
- D. Oxford Instruments Operating Manual for Model ITC4 Temperature Controller, May 1987 Issue 3, Oxford Instruments Limited, Osney Mead, Oxford OX2 0DX, England.
- E. Powder Diffraction File, Search manual, Fink Method, Inorganic, Publication SMF-26 (Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania, 1976, pp. 1017.
- F. Mineral Powder Diffraction File, Data Book (Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania, 1986) 1396 pp.
- G. VAX-PDF2/CDIF JCPDS Software Program and database on CD-ROM for VAX-VMS systems. Joint Committee on Powder Diffraction Standards, Swarthmore, Pennsylvania.
- H. The following procedures are from the Los Alamos National Laboratory Yucca Mountain Project Quality Assurance Manual in effect February, 1989.
 - H1. TWS-EES-DP-105, Thermal Calibration Procedures.
 - H2. TWS-EES-DP-101, Sample Identification and Control for Mineralogy Petrology Studies.
 - H3. TWS-EES-DP-116: Quantitative X-Ray Diffraction Data Reduction Procedure.
- I. LANL-ER-SOPs in Section 1.0, General Instructions.

8.0 RECORDS

- 8.1 All X-ray runs shall be recorded on the Daily Log, records of which shall include date of run, sample identification, analyst's signature, diffractometer used, and any unusual run conditions.
- 8.2 A catalog of all computer-automated X-ray runs shall be maintained and periodically updated and stored on magnetic or optical media.
- 8.3 All raw X-ray data stored on magnetic or optical media shall periodically be backed up onto magnetic tape and stored in a fireproof safe.

8.4 Records that are readily regenerated from the raw data such as hard copy plots and peak search data sheets may be placed in labeled three-ring binders.

9.0 ATTACHMENTS

N/A